6-05-02 1711

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Serial No. 10/046,624	Filing Date October 29, 2001	Examiner to be assigned	Group Art Unit to be assigned

Invention: "Thermoplastic Molding Compositions and Polymer Additives"

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In re Application of:	)	Group Art unit: to be assigned
AL-MALAIKA, Sahar	)	Examiner: to be assigned
Serial No.: 10/046,624	)	
Filed: October 29, 2001	)	
For: "Thermoplastic Molding Compositions and Polymer Additives"	)	Pasadena, California

06/09/02

CERTIFIED COPY OF PRIORITY DOCUMENT TRANSMITTAL LETTER

Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

Enclosed is a certified copy of United Kingdom Patent Application No. GB 9909956.6 filed April 29, 1999, needed to secure the priority dates claimed in the above-referenced Application.

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Respectfully submitted,

SHELDON & MAK

Date: June 3, 2002

By _____
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Dated 20 May 2002

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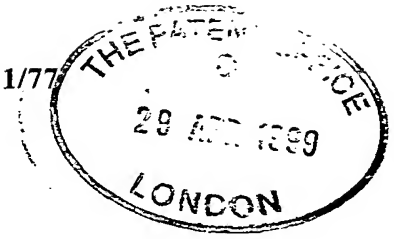

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Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

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Gwent NP9 1RH

1.	Your reference	CTE/P500075GB
2.		9909956.6 29 APR 1999
3.	Full name, address and postcode of the or of each applicant (*underline all surnames*)	ASTON UNIVERSITY ASTON TRIANGLE BIRMINGHAM B4 7ET
	Patents ADP number (*if you know it*)	6901623001
	If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM
4.	Title of the invention	THERMOPLASTIC MOULDING COMPOSITIONS AND POLYMER ADDITIVES
5.	Name of your agent (*if you have one*)	**W.P.THOMPSON & CO.**
	"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	Celcon House 289-293 High Holborn London WC1V 7HU
	Patents ADP number *(if you know it)*	158007

6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number	Country	Priority application number *(if you know it)*	Date of filing *(Day/month/year)*
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application		Number of earlier application	Date of filing *(Day/month/year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'yes' if:*
a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

THERMOPLASTIC MOULDING COMPOSITIONS AND POLYMER ADDITIVES

This invention relates to polymer additive compositions and their use. In particular it relates to an additive composition and its use for addition to thermoplastic moulding compositions comprising polyesters, such as polyethylene terephthalate.

Polyethylene terephthalate is used on a large scale for the manufacture of food packages such as bottles. Such bottles are widely utilised for packaging of beverages, such as carbonated soft drinks, beer, or mineral water. Whilst some beverage bottlers prefer clear non-pigmented bottles, others prefer coloured bottles. Particularly in the case of bottles intended for holding carbonated drinks, a sandwich construction is used in which nylon or an ethylene/vinyl alcohol resin is incorporated in a multi-layer preform with polyethylene terephthalate in order to improve the gas barrier properties of the bottles. It has also been proposed, for the same purpose, to admix a polyamide with the polyethylene terephthalate since the presence of the polyamide provides gas barrier properties.

The technique commonly used to manufacture bottles from moulding compositions comprising polyethylene terephthalate generally involves a two stage process. In the first stage granules of the moulding composition are injection moulded to make a preform. In the second stage the preform is blow moulded to the desired shape.

In such a process the polyethylene terephthalate is typically post-condensed and has a molecular weight in the region of about 25,000 to 30,000. However, it has also been proposed to use a fibre grade polyethylene terephthalate, which is cheaper but is non-post-condensed, with a lower molecular weight in the region of about 20,000. It has further been suggested to use copolyesters of polyethylene

the pigment or pigments. In addition, if the moulding composition is to be used for manufacture of food packages, the carrier must be non-toxic. Moreover the quantity of carrier used should desirably be kept to a minimum so as not to affect adversely the properties of the polyethylene terephthalate in the preform or bottle.

The softening point of polyethylene terephthalate is high. Thus a typical temperature needed for processing of polyethylene terephthalate is in the region of 260°C to 285°C. A recognised problem in the industry is that, under the high temperatures and shear conditions needed for injection moulding to make a preform and for blow moulding of the preform to make a bottle, polyethylene terephthalate tends to degrade, resulting in the formation of acetaldehyde. The presence of acetaldehyde in the material of the finished bottle is undesirable, particularly when the bottle is to be used for products for human consumption, because the acetaldehyde can migrate from the walls of the package or bottle into its contents, whereupon it adversely affects the flavour and fragrance properties of the comestible product. Although the migration of acetaldehyde from a polyethylene terephthalate bottle into a carbonated drink is undesirable, a trace of acetaldehyde can often be tolerated because the taste and fragrance of the drink are not usually noticeably affected. However, the presence of even minute amounts of acetaldehyde in a non-carbonated drink, such as still mineral water, tends to impart a most undesirable adverse taste and odour to the drink.

Methods for measurement of acetaldehyde in industrially injection-moulded polyethylene terephthalate preforms have been described by F. Villain *et al.*, Journal of Polymer Science, Vol. 52, 55-60 (1994).

Attempts have been made by equipment manufacturers to

acid, 5-hydroxyisophthalic acid, 3,5-dihydroxybenzoic acid, phenyl isocyanate, phthalic anhydride, 4-aminobenzoic acid, resorcinol, and diphenylamine. They reported that, when used in an injection machine at a weight percentage of 1% based upon the weight of polyethylene terephthalate, 4-aminobenzoic acid, 3,5-dihydroxybenzoic acid and diphenylamine were found to be the most effective additives under laboratory conditions. These authors further postulated that 4-aminobenzoic acid acts as both a free radical scavenger and a hydroxyethyl chain blocker.

United States Patent No. 5,258,233 describes polyester/polyamide blends which have gas barrier properties and through reduction of acetaldehyde concentration in the polyester improve the storage properties of foodstuffs over previously reported blends. The use of low molecular weight partially aromatic polyamides having a number average molecular weight of less than 15,000 or low molecular weight aliphatic polyamides having a number average molecular weight of less than 7,000 is said to be more effective in reducing residual acetaldehyde in polyethylene terephthalate based polyesters than high molecular weight polyamides. However, a recognised problem associated with utilising a polyamide as an additive in polyethylene terephthalate formulations is that it causes discolouration of the preform due to degradation during the melt extrusion process.

International Patent Publication No. WO 94/29378 teaches a polyester/zeolite admixture which is said to have an excellent gas barrier property and an improved flavour retaining property as well as clarity. Addition of small- or medium-pore zeolites in a critical amount to a polyester is said to reduce the concentration of acetaldehyde in the polyester without producing haze.

In International Patent Publication No. WO 98/18848

succinic anhydride or phthalic anhydride. The use of pyromellitic anhydride for end capping of polyethylene terephthalate has been proposed in United States Patent No. 5,243,020.

European Patent Publication No. 0 878 502 A discloses a stabiliser mixture for thermally stabilising organic polymers, especially food packages, consisting of (1) α-tocopherol, (2) a solid polyhydroxy compound which is selected from the group consisting of triglycerin, ditrimethylolpropane, dipentaerythritol, tripentaerythritol, D-mannitol, D-sorbitol, and xylitol or (3) an acid binding material or a mixture of components (2) and (3). Amongst the polymers which can be thermally stabilised in this way are said to be polyesters, including polyethylene terephthalate. However, no experimental evidence is provided which involves use of polyethylene terephthalate.

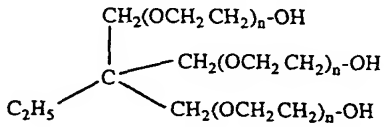
There is a need to provide a polymer additive for incorporation in moulding compositions which comprise polyethylene terephthalate, a copolyester thereof, or a blend of one of these with a polyamide, in order to reduce the amount of acetaldehyde formed during processing of such moulding compositions.

There is a further need to provide a polymer additive which does not lead to discolouration or haze when polyester moulding compositions which consist of or contain polyethylene terephthalate or a copolymer thereof and which contain the polymer additive are subjected to injection moulding and/or blow moulding.

There is a still further need to provide a process for production from moulding compositions containing polyethylene terephthalate of blow moulded articles, such as bottles and preforms therefor, which will not release significant quantities of acetaldehyde after formation.

those in which a pair of hydroxy groups are attached to respective carbon atoms which are separated one from another by a single carbon atom.

As examples of suitable hydroxylic compounds there can be mentioned diols such as ethylene glycol, propane-1,2-diol, propane-1,3-diol, butane-1,4-diol, pentane-1,5-diol, hexane-1,2-diol, 2-methylpentane-2,4-diol, 2,5-dimethyl-hexane-2,5-diol, cyclohexane-1,2-diol, cyclohexane-1,1-dimethanol, diethylene glycol, triethylene glycol, and polyethylene glycols having, for example, a molecular weight from about 800 to about 2000, such as Carbowax™ 1000 which has a molecular weight of about 950 to about 1050 and contains from about 20 to about 24 ethyleneoxy groups per molecule; triols, such as glycerol, trimethylolpropane, 2,3-di-(2'-hydroxyethyl)-cyclohexan-1-ol, hexane-1,2,6-triol, 1,1,1-tris-(hydroxymethyl)ethane, 3-(2'-hydroxyethoxy)-propane-1,2-diol, 3-(2'-hydroxypropoxy)-propane-1,2-diol, 2-(2'-hydroxyethoxy)-hexane-1,2-diol, 6-(2'-hydroxypropoxy)-hexane-1,2-diol, 1,1,1-tris-[(2'-hydroxyethoxy)-methyl]-ethane, 1,1,1-tris-[(2'-hydroxypropoxy)-methyl]-propane, 1,1,1-tris-(4'-hydroxyphenyl)-ethane, 1,1,1-tris-(hydroxyphenyl)-propane, 1,1,3-tris-(dihydroxy-3-methylphenyl)-propane, 1,1,4-tris-(dihydroxyphenyl)-butane, 1,1,5-tris-(hydroxyphenyl)-3-methylpentane, trimethylolpropane ethoxylates of the formula:

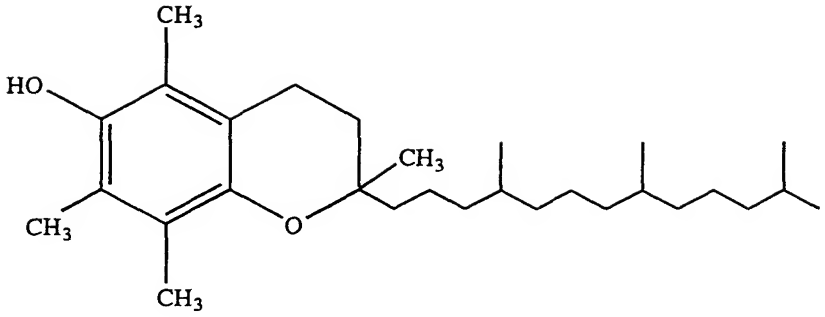


in which n is an integer, or trimethylolpropane propoxylates of the formula:

selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydroxylic compounds containing at least two hydroxy groups, and subjecting said thermoplastic moulding composition to an injection moulding step thereby to form a moulded article. Such a moulded article can be a preform for use in a subsequent blow moulding step to form a bottle.

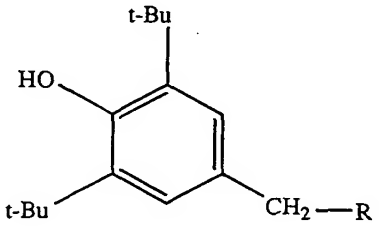
The invention also provides the use of a hydroxylic compound selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydroxylic compounds containing at least two hydroxy groups as an additive to a thermoplastic moulding composition comprising a polymer component comprising polyethylene terephthalate or a copolyester thereof for the reduction of the amount of acetaldehyde formed upon subjecting said moulding composition to melt processing.

By following the teachings of the invention it is possible to produce a thermoplastic polyester material with excellent reduction in levels of aldehyde without producing any discolouration of the material upon processing thereof. Thus it is possible to produce preforms and bottles of excellent clarity and lack of haze or colour by injection moulding a polymer composition containing polyethylene terephthalate or a copolyester thereof, optionally in admixture with a polyamide, and a hydroxylic compound selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydroxylic compounds containing at least two hydroxy groups, so as to form a preform and thereafter blow moulding the

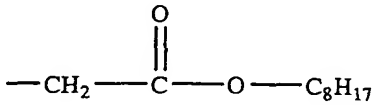


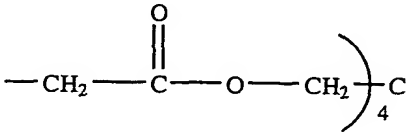
This compound occurs in nature and is known as Vitamin E. The naturally occurring compound has the R,R,R chiral configuration. It is, however, available in synthetic form as d,l-α-tocopherol. Preferably the synthetic form, d,l-α-tocopherol, is used. The synthetic form has 8 isomers each having a different chiral configuration.

Other hindered phenol antioxidants which can be considered for use in the present invention are those of the general formula:

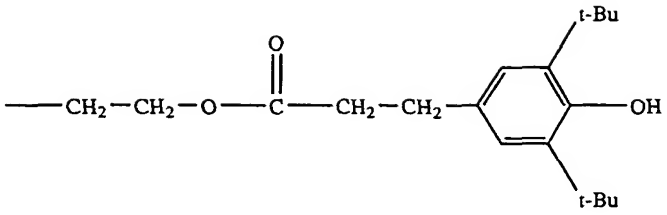


in which R is hydrogen,

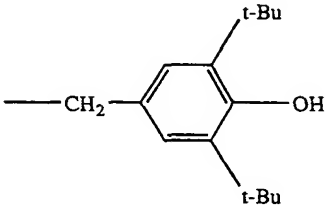




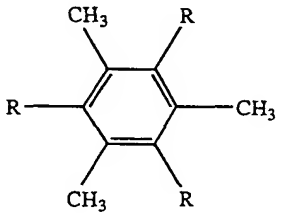
where R is



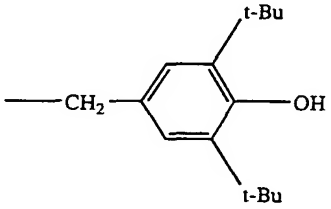
or



Another type of hindered phenol antioxidant which can be considered for use in the present invention is exemplified by compounds of the formula:

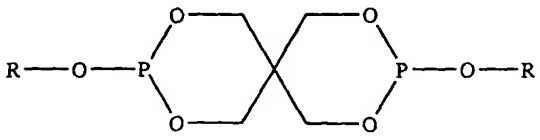


in which R is

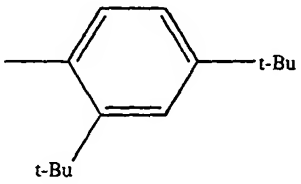


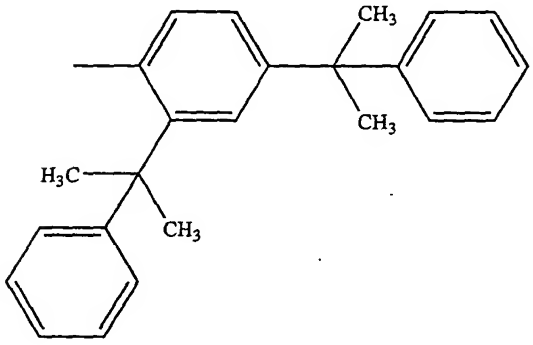
or

Phosphite antioxidants are another class of antioxidants that can be used, for example, those of the formula:



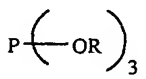
in which R is



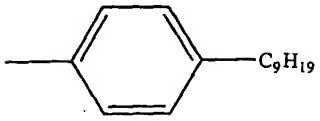


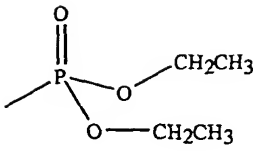
or $C_{18}H_{37}$.

Other phosphite antioxidants include those of the formula



in which R represents a group of the formula:





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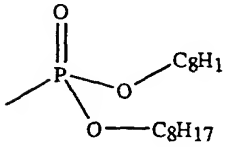


The advantage of using α-tocopherol is that, not only is it an extremely efficient antioxidant, but also it is non-toxic. Thus it is an extremely suitable material to use in a packaging material that is to be used for packaging bottled drinks and other foodstuffs.

A mixture of two or more antioxidants of the same or different type can be used, if desired. For example, it is possible to use a mixture of a hindered phenol type of antioxidant and a phosphite type of antioxidant.

It is preferred that in the polymer additive of the invention the hydroxylic compound:liquid carrier weight ratio ranges from about 0.1:1 to about 3:1. Preferably this ratio is from about 0.5:1 to about 1.5:1.

The amount of hydroxylic compound to be used in the thermoplastic moulding composition can vary within wide limits but typically ranges from about 0.0001 % by weight up to about 2 % by weight based upon the weight of the polymer component, i.e. the polyester or copolyester or blend thereof with a polyamide. More preferably the amount of hydroxylic compound used ranges from about 0.01 % by weight up to about 1 % by weight based upon the polymer component.

The amount of antioxidant or mixture of antioxidants, when used in the thermoplastic moulding composition, can vary within wide limits but typically ranges from about 0.0001 % by weight up to about 2 % by weight based upon the weight of

undiluted form. In this case no organic liquid carrier is used.

The invention is further illustrated in the following Examples in which all parts are by weight, unless otherwise specified.

Examples 1 to 11

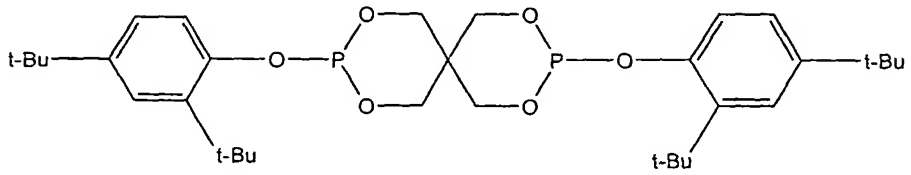
A series of polymer additive mixtures was made up by mixing the ingredients listed in Table 1 in the ratio of the listed amounts. Each mixture was then used in conjunction with the specified weight of a granular polyethylene terephthalate (PET) moulding composition to form bottle preforms with a weight of 46.9 g. A comparison preform was prepared on the same occasion under identical conditions in each case, except that the additive mixture under test was omitted.

In each of Examples 1 to 11 the polyester was extruded in the presence of the additive mixture in an injection moulding machine having two cavities using a mould temperature of 283°C, a barrel temperature of 278°C at the nozzle end and 275°C in the rest of the barrel, an injection pressure of 100 bar and a cycle time of 38.5 s.

The acetaldehyde content of the resulting preforms was determined by the liquid/gas headspace chromatography technique described by F. Villain *et al.*, Journal of Polymer Science, Vol. 52,

Notes:

1. U-626 is a bis-phosphite of pentaerythritol having the formula:



2. Clearslip™ 2 is an oil-based carrier available from ColorMatrix Europe Ltd, of Units 9-11 Unity Grove, Knowsley Business Park, Merseyside, L34 9GT.

Examples 12 and 13

The same general procedure was followed in these Examples as in Examples 1 to 11, except that trimethylol-propane was replaced by dipentaerythritol (DPE) and tripentaerythritol (TPE) respectively. The results are set out below in Table 2.

Examples 14 to 16

The same general procedure was followed as in Examples 1 to 11, except that the bottle preforms weighing 28 g each were extruded using a commercial injection moulding machine having 48 cavities. The injection pressure was 100 bar, while the barrel temperature ranged from 295°C at its nozzle end to 285°C at its inlet end, with intermediate parts of the barrel being at 279°C. The results are set out below in Table 3. Examples 14 and 15 used Clearslip™ 2 while Example 16 used Clearslip™ 3.

Examples 17 to 20

In these Examples various commercial injection moulding machines were used to make bottle preforms under conditions similar to those described above in relation to Examples 14 to 16. The results are summarised in Table 4 below.

Examples 21 to 35

In these Examples the same general procedure was used as was used in Examples 1 to 11.

The results are set out below in Table 5.

CLAIMS:

1. A polymer additive for addition to a thermoplastic moulding composition comprising polyethylene terephthalate or a copolyester thereof so as to effect reduction of the level of acetaldehyde resulting after processing thereof, said polymer additive comprising a hydroxylic compound selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydoroxylic compounds containing at least two hydroxy groups, uniformly distributed in a polyester-compatible organic liquid carrier.

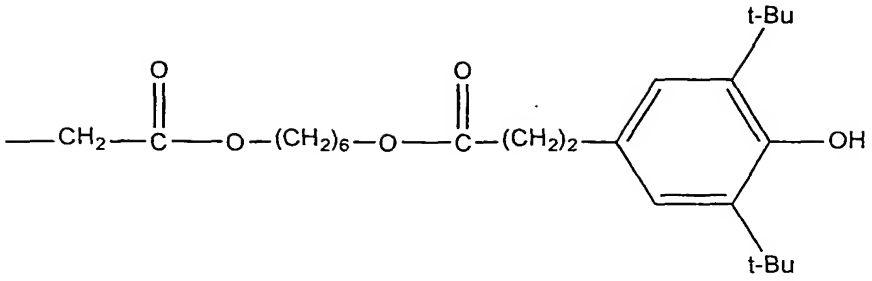
2. A polymer additive according to claim 1, in which in the hydroxyl compound contains a pair of hydroxy groups attached to respective carbon atoms which are separated one from another by a single carbon atom.

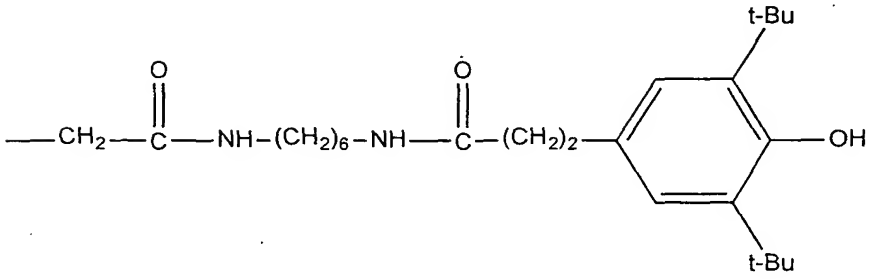
3. A polymer additive according to claim 1 or claim 2, in which said hydroxylic compound is selected from triglycerin, trimethylolpropane, dipentaerythritol, tripentaerythritol, D-mannitol, D-sorbitol, and xylitol.

4. A polymer additive according to any one of claims 1 to 3, in which the hydroxylic compound:liquid carrier weight ratio ranges from about 0.1:1 to about 1.5:1.

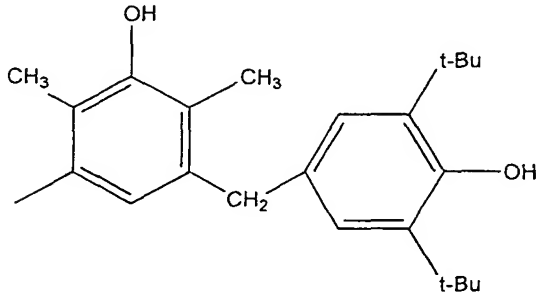
5. A polymer additive according to any one of claims 1 to 4, in which the polyester-compatible organic liquid carrier is an oil-based vehicle.

6. A polymer additive according to any one of claims 1 to 5, further comprising at least one polyester-compatible





or



11. A polymer additive according to claim 9, in which the antioxidant comprises synthetic Vitamin E.

12. A polymer additive according to any one of claims 1 to 12, further comprising a phosphite antioxidant.

13. A polymer additive according to claim 12, in which the phosphite antioxidant has the structure:

at least two hydroxy groups, and cycloaliphatic hydoroxylic compounds containing at least two hydroxy groups.

16. A thermoplastic moulding composition according to claim 15, in which the amount of said hydroxylic compound ranges from about 0.0001 % by weight up to about 1 % by weight based upon the weight of the polymer component.

17. A thermoplastic moulding composition according to claim 15 or claim 16, in which the hydroxylic compound contains a pair of hydroxy groups attached to respective carbon atoms which are separated one from another by a single carbon atom.

18. A thermoplastic moulding composition according to any one of claims 15 to 17, in which said hydroxylic compound is selected from triglycerin, trimethylolpropane, dipentaerythritol, tripentaerythritol, D-mannitol, D-sorbitol, and xylitol.

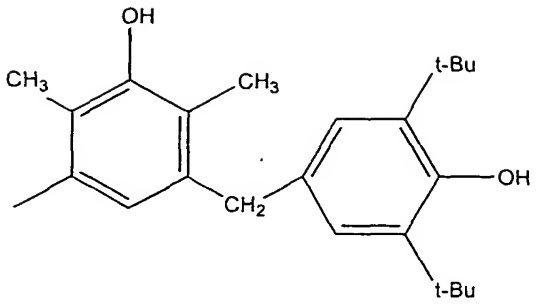
19. A thermoplastic moulding composition according to any one of claims 15 to 18, in which the amount of hydroxylic compound ranges from about 0.0001 % by weight up to about 1 % by weight based upon the weight of the polymer component.

20. A thermoplastic moulding composition according to any one of claims 15 to 19, further comprising an antioxidant.

21. A thermoplastic moulding composition according to claim 20, in which the antioxidant is a hindered phenol antioxidant.

22. A thermoplastic moulding composition according to claim 21, in which the antioxidant is a 4-substituted-2,6-di-

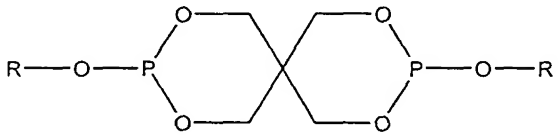
or



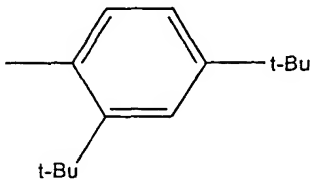
24. A thermoplastic moulding composition according to claim 22, in which the antioxidant comprises synthetic Vitamin E.

25. A thermoplastic moulding composition according to any one of claims 15 to 24, in which the antioxidant comprises a phosphite antioxidant.

26. A thermoplastic moulding composition according to claim 25, in which the phosphite antioxidant has the structure:



in which R is



30. A process according to claim 28 or claim 29, in which the hydroxylic compound contains a pair of hydroxy groups attached to respective carbon atoms which are separated one from another by a single carbon atom.

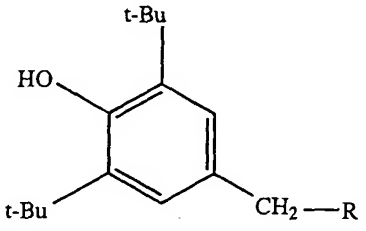
31. A process according to any one of claims 28 to 30, in which said hydroxylic compound is selected from triglycerin, trimethylolpropane, dipentaerythritol, tripentaerythritol, D-mannitol, D-sorbitol, and xylitol.

32. A process according to any one of claims 28 to 31, in which the thermoplastic moulding composition further comprises an antioxidant.

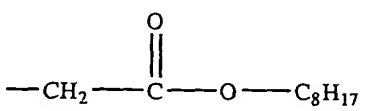
33. A process according to claim 32, in which the antioxidant is a hindered phenol antioxidant.

34. A process according to claim 33, in which the antioxidant is a 4-substituted-2,6-di-tertiary butyl phenol or an α-tocopherol.

35. A process according to claim 33, in which the antioxidant has the formula:



in which R is hydrogen,

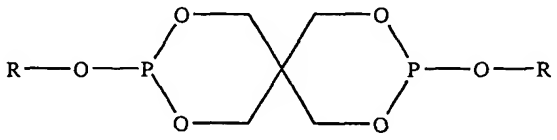


which the thermoplastic moulding composition further comprises at least one polyester-compatible colorant.

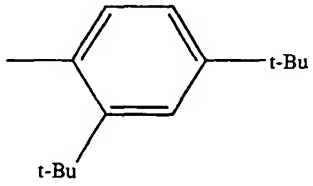
38. A process according to any one of claims 28 to 37, in which the thermoplastic moulding composition further comprises a minor amount of a liquid carrier for said aliphatic hydroxylic compound.

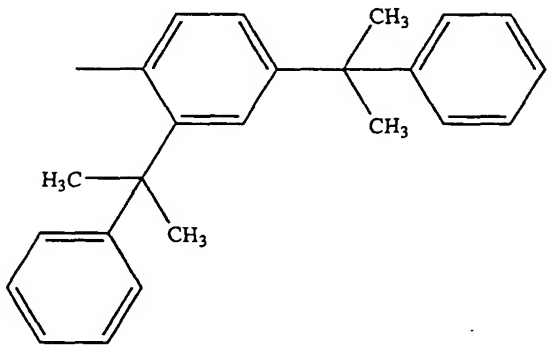
39. A process according to any one of claims 28 to 38, in which the antioxidant comprises a phosphite antioxidant.

40. A process according to claim 39, in which the phosphite antioxidant has the structure:



in which R is





or $C_{18}H_{37}$.

ABSTRACT

THERMOPLASTIC MOULDING COMPOSITIONS AND POLYMER ADDITIVES

A polymer additive is described for addition to a thermoplastic moulding composition comprising polyethylene terephthalate or a copolyester thereof so as to effect reduction of the level of acetaldehyde resulting after processing thereof, said polymer additive comprising a hydroxylic compound selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydroxylic compounds containing at least two hydroxy groups, uniformly distributed in a polyester-compatible organic liquid carrier. The invention further relates to the use of a hydroxylic compound selected from aliphatic hydroxylic compounds containing at least two hydroxy groups, aliphatic-cycloaliphatic compounds containing at least two hydroxy groups, and cycloaliphatic hydoroxylic compounds containing at least two hydroxy groups, as an additive to a thermoplastic moulding composition for the reduction of the amount of acetaldehyde formed upon subjecting said moulding composition to melt processing. Thermoplastic moulding compositions, processes using same and preforms and bottles made therefrom are also described. Preferred hydroxylic compounds include triglycerin, trimethylolpropane, dipentaerythritol, tripentaerythritol, D-mannitol, D-sorbitol, and xylitol.